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# BORON FILAMENTS



*by Robert M. Witucki*



Prepared under Contract No. NASw-652 *by*
ASTRO RESEARCH CORPORATION
Santa Barbara, Calif.
*for*



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## I. INTRODUCTION

The combination of properties of elemental boron filaments make these unique among all other continuous filamentary materials. The presently known filament characteristics indicate great potential for many special applications pertaining to space flight, as well as for the development of new composite materials. The properties of particular interest include the very high modulus of elasticity, about 55 x $10^6$ psi, the high melting temperature, about 2040°C, the low density, about 2.35 g/cc, and the considerable retention of strength at temperatures to 1000°C. Although present strength-to-weight ratio for boron filaments is similar to that for glass filaments there is real potential for increasing the boron filament strength significantly.

Possible applications of boron filaments include: the positive deployment of large radio wave reflective satellites or other lightweight structures, using stored elastic energy ~~(Ref. 1)~~; use in deployable re-entry structures where a high strength-to-weight ratio is required at a high temperature ~~(Ref. 2)~~; and in the form of metal-boron composites for compressively loaded shell and column structures, such as are used in pressure resistant submarine hulls and atomic reactor supports ~~(Ref. 3)~~.

## II. FILAMENT FABRICATION

Since boron filaments are not readily available for experimentation, an apparatus was assembled to make laboratory quantities for evaluation.

In general aspects the continuous laboratory preparation process is similar to one described by Claude P. Talley, et al (Ref. 4) of Texaco Experiment, Inc. A 1/2 mil diameter tungsten wire is used as a substrate on which the boron is deposited. The wire is first cleaned by heating in a cleaning chamber which contains a flowing hydrogen atmosphere. This chamber contains a liquid mercury seal at each end through which the wire is passed. The mercury also provides electrical contacts with the wire, and a current is passed through the wire to heat it to about 1200°C while in the cleaning chamber. The wire then passes directly into a second similar chamber where boron deposition is accomplished. The wire is independently heated in the coating chamber to a temperature in the range of 1000 to 1200°C. A mixture of boron tribromide vapor and hydrogen is passed through the coating chamber. The $BBr_3$-$H_2$ mixture reacts at the heated tungsten surface and deposits elemental boron in a non-crystalline form. It is essential that the wire temperature be limited to about 1200°C since at higher temperatures the boron begins to crystallize with a resulting great decrease in tensile strength of the filament.

The growth of the boron filaments results from a surface reaction which does not occur at the same rate at all points on the surface. In fact, the surface must also exert a catalytic effect for the reaction to occur, since growth occurs readily on metal wires but apparently not on carbon filaments.

A photograph of the initial boron coating apparatus used is shown in Figure 1. The 1/2 mil tungsten wire enters the apparatus at the bottom, and the boron filament is taken up at the top. This photograph was taken during an actual boron coating run and shows the temperature distribution along the filament in both the cleaning and coating chambers. Subsequent improvements have been made to the apparatus to permit longer time operation, and included automatic recycling of unreacted boron bromide.

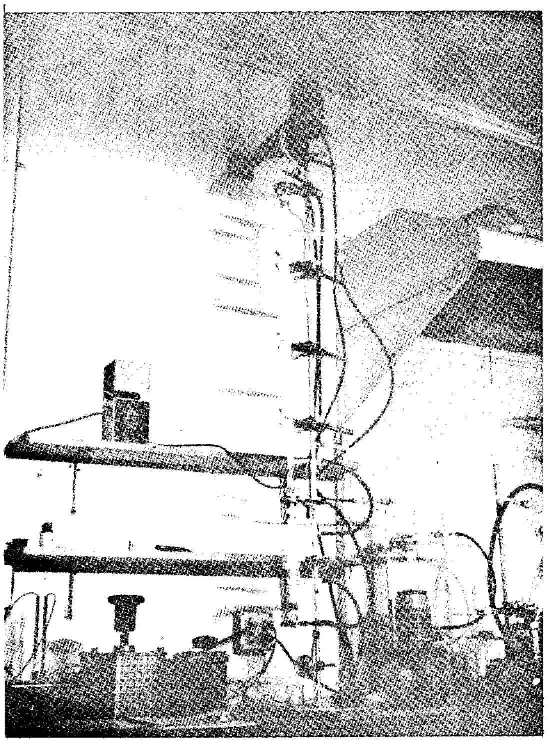

Figure 1
Boron Filament Coating Apparatus

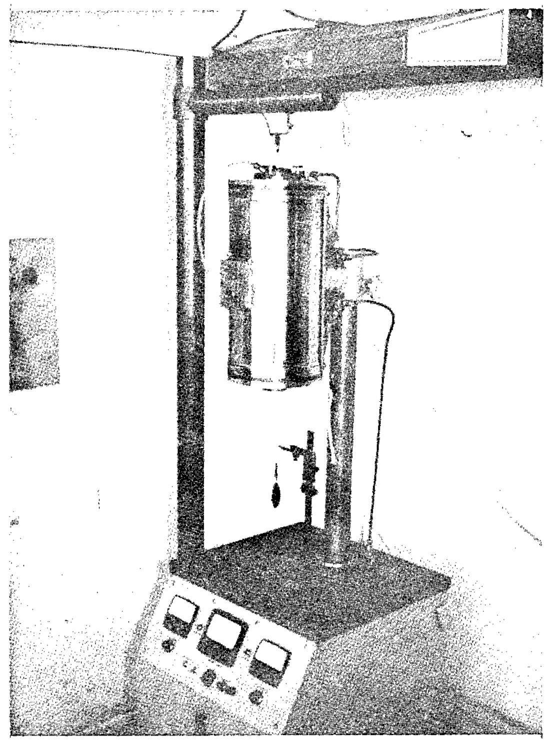

Figure 2
ASTRO Model 2570 High Temperature
Filament Test Apparatus

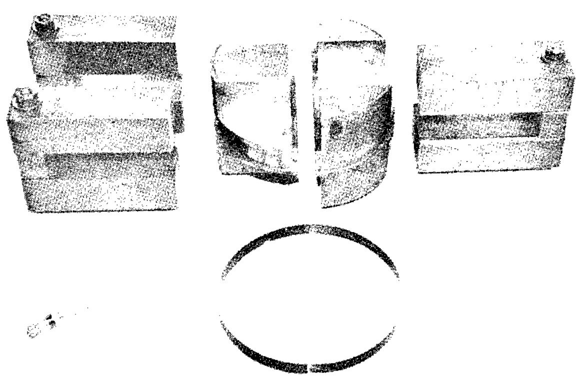

Figure 3
Boron Filament Test Band and
Split Ring Test Apparatus

## III. TENSILE STRENGTH OF BORON FILAMENTS

Considerable variations have been observed in the tensile strength of individual boron filaments produced. For the most part these have varied from 25,000 for filaments with flaws to 300,000 psi, with some individual readings higher for filaments produced under optimal conditions. Filaments have been tested both at room temperature and at elevated temperatures to 1200°C. For the purpose of these tests, filaments were bonded to loading tabs and tensile loads applied by calibrated weights. The setup for high temperature tests, using an ASTRO Model 2570 furnace in vertical position with muffle tube insert, is shown in Figure 2.

In order to obtain useful values of strength from individual filaments, it is necessary to test many filaments, and to treat the results statistically. This is both tedious and time consuming, and the results can characterize principally the filament selection process. The average strength of a long length of filament can, however, be characterized from data on test bands, and can yield a more meaningful engineering figure. For these reasons test bands 4 inches in diameter and 0.3 inches wide were wound with several layers of boron filaments, each layer with 100 turns. A room temperature setting epoxy resin binder was applied during the winding process. The boron filament varied from 1.8 to 2.0

mils. Bands were then stressed to failure using a split ring test apparatus shown in Figure 3. The load at failure for a five layered (500 turn) band was 0.68 pounds per filament. This corresponds to a single filament stress of 216,000 psi assuming all 2 mil diameter filaments, or to 268,000 psi assuming all 1.8 mil diameter filaments. The value of specific strength measured for this test band was $1.14 \times 10^6$ inches; however, this value is unnecessarily low because of resin excess resulting from the use of only relatively few turns of the filament.

## IV. MICROSCOPIC EXAMINATION OF FILAMENTS

The filaments produced have a characteristic surface appearance. This is shown under several magnifications in Figures 4 through 6.

Figure 4 shows the characteristic nodular surface of a vapor deposited coating. Under some coating conditions one nodule may begin to grow at a much greater rate than neighboring ones, and a spike is generated, while occasionally several spikes will originate immediately adjacent to each other. This condition results in a mechanically weak point in the filament. Figures 5 and 6 show similar surfaces under greater magnification. Figure 6 is, however, remarkable because it shows what is apparently a straight line crack in a filament as produced. The apparent irregularities in the observed line appear to be the intersection of a straight line crack with the rough nodular surface.

What is apparently a similar phenomenon, but enhanced by etching, is shown in Figures 7 and 8. The spiral groove shown in Figure 7 was traced along the filament for 7 complete revolutions. Similar grooves have been observed on other filaments after etching. In every case, a left hand spiral was observed (as receding from the observer). This is also true for the non-etched crack shown in Figure 6. The crack as shown in Figure 7 is about 0.46

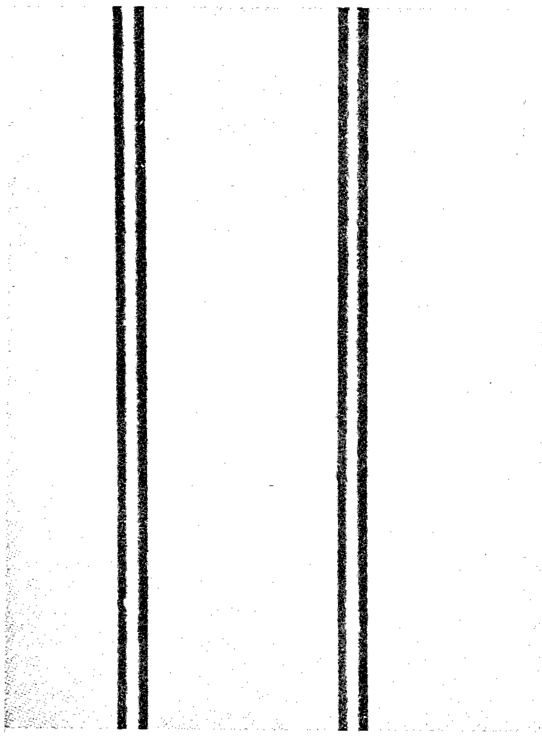

Figure 4
Boron Filaments, 3.1 Mil
Diameter, About 50X

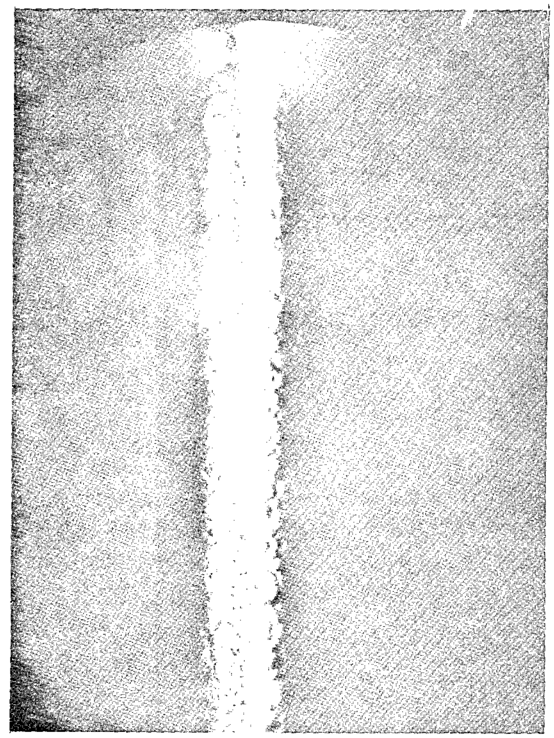

Figure 5
Boron Filament, About 320X


Figure 6
Boron Filament, About 1000X

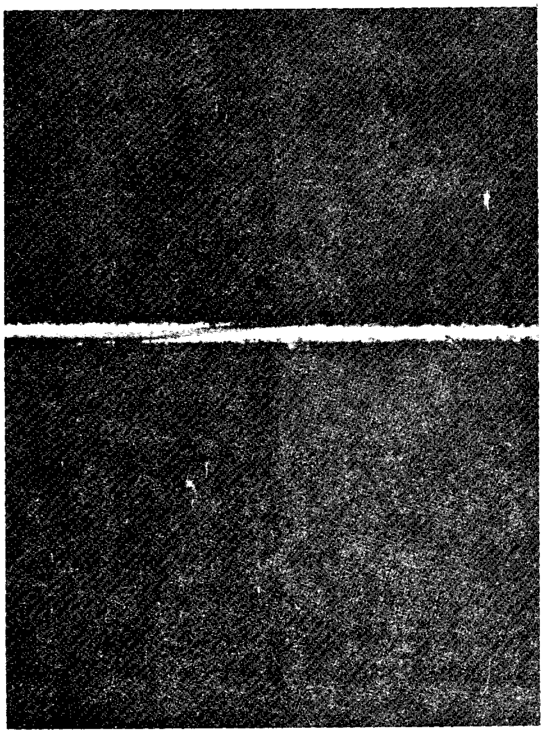

Figure 7
Boron Filament Showing Etched Spiral Crack, About 90X

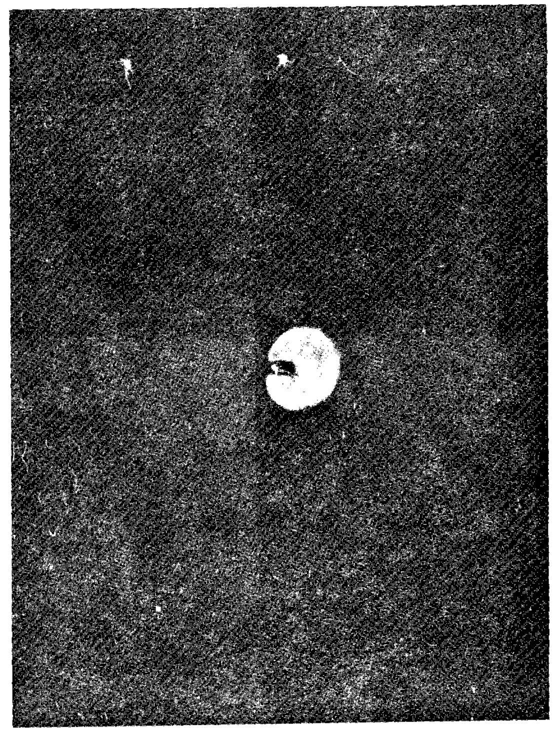

Figure 8
Boron Filament Showing Etched Crack, End View, About 150X

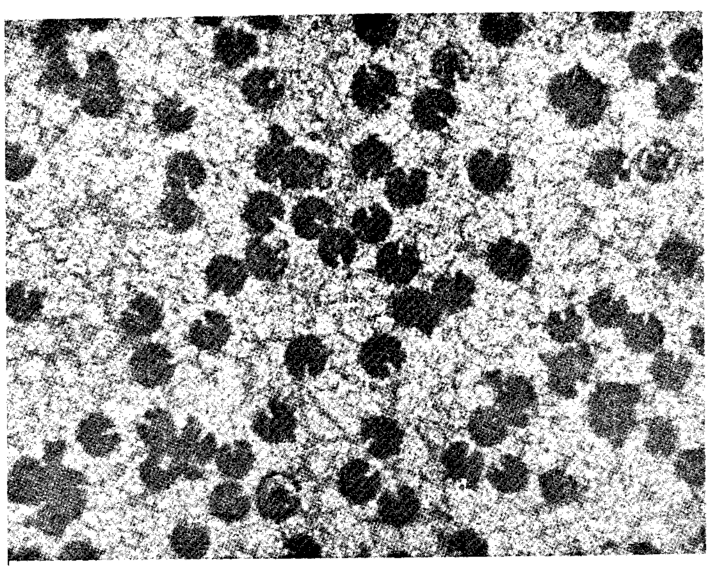

Figure 9
Boron Filament - Aluminum Composite, About 90X

mils wide by 1 mil deep. In depth, therefore, it extends down to the original tungsten wire core. The particular filament shown in these figures was loaded to failure in tension before etching, and showed a tensile strength of only 60,000 psi. The low strength can be associated with the great surface irregularity that can be observed in Figure 7.

The fact that a crack has been observed in a filament as produced, shown in Figure 6, indicates that these can develop during fabrication. Such cracks can be interpreted as evidence for considerable strain developed between the filament core, which has been converted to a tungsten boride during coating, and the exterior sheath of non-crystalline boron. Additional evidence for this is given subsequently.

## V. BORON FILAMENT-ALUMINUM COMPOSITES

Two initial boron-aluminum composites have been made. In the first a 24ST aluminum alloy was melted and poured into an evacuated 1/8 inch diameter Vycor tube packed with boron filaments. The boron filaments were preheated before infiltrating with aluminum. Sections of the composite were prepared for microscopic examination, and a typical section shown in Figure 9. It can be seen in Figure 9 that a large percentage of the filaments have split, forming regular V-shaped gaps in the filaments. Further, it appears that aluminum has infiltrated into these splits. These splits have not developed as a result of grinding the section, since similar splits have been observed on filaments exposed at a fractured surface.

A second composite was made, without preheating the boron filaments, by quickly immersing a Vycor tube packed with boron filaments into a melt of aluminum. The tube was then quickly evacuated to draw the molten aluminum into the tube. Figure 10 shows a characteristic photomicrograph of a section prepared by grinding with 600 grit silicon carbide. It can be seen that this composite does not show the V-shaped splits seen previously. However, nearly every filament end in the figure shows either three or four radial cracks extending outward from the center.

These characteristic cracks are shown in larger magnification in Figure 11.

In contrast to the "V" cracks shown in Figure 8, the splitting of the filaments shown in Figures 9 and 10 appears to be a result of the grinding steps required for preparing a section for microscopic observation. Similar splitting has been observed in a section prepared from boron filaments embedded in a room temperature setting resin. In this case the filaments were not heated after fabrication, and heating therefore cannot be a cause. It therefore appears that rolling of the abrasive grains over the filament ends during grinding must initiate these fractures, and that the fractures serve to release strain developed between the tungsten boride core and the boron sheath of the filament.

An interesting view of the difference in properties of the boron sheath and the core can be seen in Figure 12. It can be seen that the 600 grit silicon carbide has selectively ground both the aluminum matrix and the tungsten boride core of the filaments.

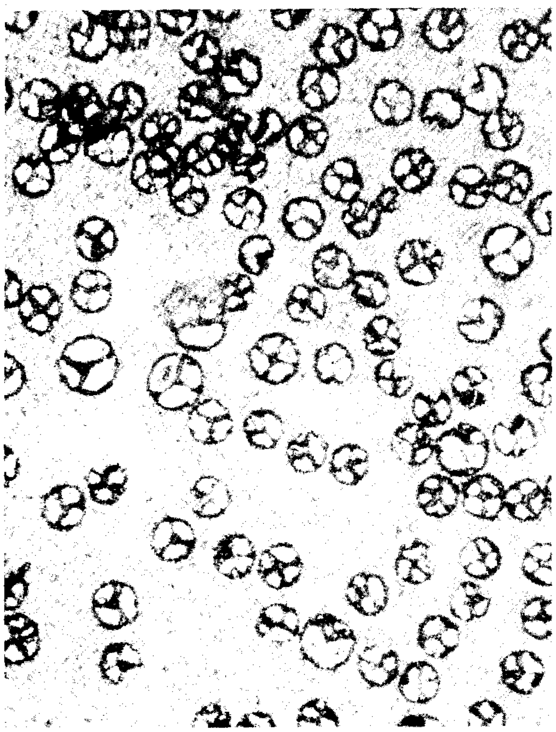

Figure 10
Boron Filament - Aluminum
Composite, About 90X

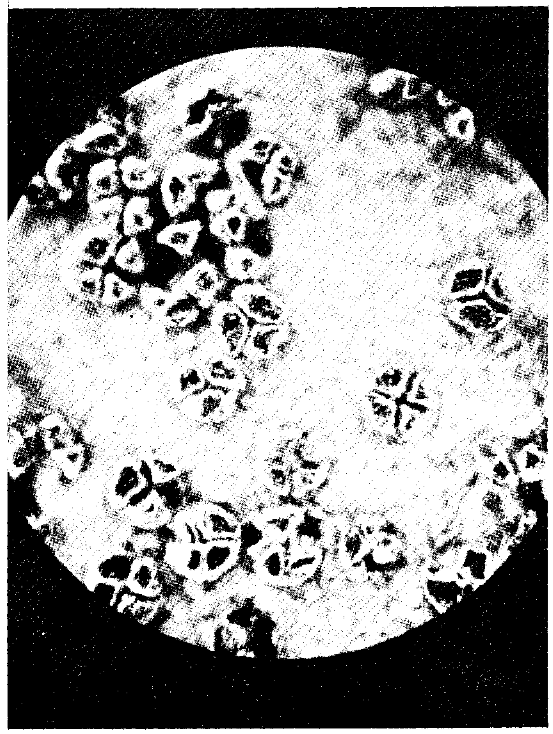

Figure 11
Boron Filament - Aluminum
Composite, About 175X

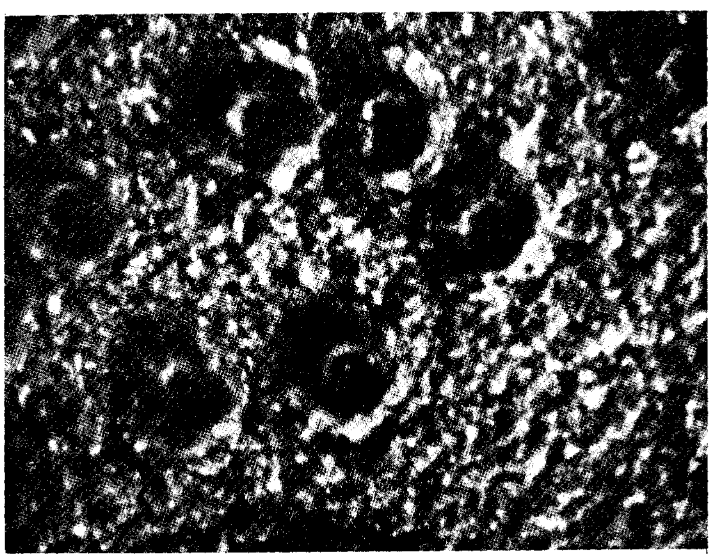

Figure 12
Boron Filament - Aluminum
Composite, About 300X

## VI. SUMMARY AND CONCLUSIONS

Results of the work to date show that filaments made by depositing elemental boron on tungsten wire are under considerable internal strain. This effect can arise in two ways: First, from expansion of the tungsten lattice as boron is introduced. This effect could cause the observed spiral cracks, as well as the radial end cracks that develop on grinding end sections. Second, since the filaments are formed at a high temperature a differential thermal expansion between the tungsten boride core and the boron sheath can cause significant stresses to develop on cooling.

Such internal stresses can drastically modify the inherent strength of both the boron sheath and the tungsten boride core. The presence of cracks as observed in the filaments may increase the overall strength of the filaments by relieving the internal stresses developed during fabrication. However, if cracks are present oriented approximately normal to the filament axis these would act as stress raisers and significantly weaken the filaments. In either event, using a core material with characteristics different from the tungsten boride can be expected to have a great effect on the mechanical properties of the composite filament.

It will be necessary to evaluate all of the above possibilities in detail before the real strength of the amorphous boron sheath can be evaluated, and before this can be compared with the theoretical strength of several million psi which one might expect

based on the high modulus of elasticity. At the same time the value of 220,000 to 270,000 psi found for the 500 turn test band, made with little development effort, is highly encouraging, and is of interest even at this level. With this result specific applications for boron filaments should be carefully evaluated.

At the same time, however, the basic limitations of the present process must be considered. These principal limitations include the cost of the tungsten core, as well as the limited lengths that can be drawn, and the very slow production rates for the boron coating process.

## VII. RECOMMENDATIONS FOR FUTURE WORK

Experimental work should be performed to gain an understanding of internal stress conditions within the tungsten-boron composite filaments. It will not be possible to evaluate the ultimate potential and possible limitations of such filaments until such an understanding has been achieved.

More detailed engineering data should be obtained for the best filaments that can be produced reproducibly today. This should include more data on assembled samples, including test bands, as well as more elevated temperature data on single filaments.

Exploratory studies should be carried out on the possible use of other substrates for boron deposition, or on the possibility of continuous filament formation from a melt or solution.

An exploratory study should be made of boron filament-metal composites to establish the general characteristics of these, so that the future potential of these can be realistically judged.

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